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(54) DUST CORE AND ITS MANUFACTURING METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To realize such a dust core that can keep a magnetic characteristic even when it is used in a high-temperature environment and shows no reduction of strength or no dimensional change, and can endure cutting or the like.

SOLUTION: This dust core is formed by joining an iron powder or an iron powder whose surface is covered with a phosphate compound film, with a resin. The joint resin is made of polyphenylene sulfide or thermoplastic polyimide, and the content of resin is 0.15 to 1 mass percentage of total mass.

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CLAIMS

[Claim(s)]

[Claim 1] It is the dust core which said resin for association is in any of polyphenylene sulfide and thermoplastic polyimide in the dust core which combined iron powder or the iron powder which gave the phosphoric acid compound coat to the front face by resin, and is characterized by what the resin content is 0.15 - 1 mass % of total mass.

[Claim 2] It is the dust core which said resin for association is the mixture of polyphenylene sulfide and thermoplastic polyimide in the dust core which combined iron powder or the iron powder which gave the phosphoric acid compound coat to the front face by resin, and is characterized by what the resin content is 0.15 - 1 mass % of total mass.

[Claim 3] It is the dust core which said resin for association is in the dust core which combined iron powder or the iron powder which gave the phosphoric acid compound coat to the front face by resin in any of mixture with the resin with a glass transition temperature higher than the mixture or thermoplastic POIMIDO, and said thermoplastic polyimide with high resin of glass transition temperature from polyphenylene sulfide (henceforth PPS), and said PPS, and is characterized by what the resin content is 0.15 - 1 mass % of total mass.

[Claim 4] Said resin for association is a dust core which polyphenylene sulfide (henceforth PPS), thermoplastic polyimide, and glass transition temperature are mixture with resin higher than said PPS at least in the dust core which combined iron powder or the iron powder which gave the phosphoric acid compound coat to the front face by resin, and is characterized by what the resin content is 0.15 - 1 mass % of total mass.

[Claim 5] The dust core according to claim 3 or 4 in which resin with a glass transition temperature higher than said thermoplastic polyimide is in any of non-thermoplasticity polyimide, polyamidoimide, and poly amino bismaleimide, and resin with a glass transition temperature higher than said PPS is in any of the resin mentioned above as what has a glass transition temperature higher than polyphenylene oxide, Pori Sall John, polyether sulfone, polyarylate, polyether imide, and said thermoplastic polyimide.

[Claim 6] The dust core given in any of claims 3-5 they are whose content of the resin with a glass transition temperature higher than the high resin or said thermoplastic polyimide of glass transition temperature from said PPS is below one half among the total content 0.15 of the resin for association - 1 mass %.

[Claim 7] The manufacture approach of the dust core characterized by heat-treating while doing 0.15-1 mass % mixing of which the resin powder for association given in (6) with a mass ratio from following (1) and pressing this mixed powder into iron powder or the iron powder which gave the phosphoric acid compound coat to the front face.

(1) Polyphenylene sulfide (henceforth PPS)

(2) thermoplastic polyimide (3) -- the mixture (4) of said PPS and thermoplastic polyimide -- mixture (5) with resin with a glass transition temperature higher than said PPS and said PPS -- mixture (6) with resin with a glass transition temperature higher than said thermoplastic polyimide and said thermoplastic polyimide -- said PPS and said thermoplastic polyimide, and glass transition temperature -- mixture [claim 8] with resin [at least] higher than said PPS The manufacture approach of the dust core characterized by heat-treating while it dries after mixing the liquid which dissolved which resin powder for association of (1) - (6) according to claim 7 in the organic solvent, and considering as the resin covering iron powder said whose resin content is 0.15 - 1 mass % in a mass ratio and pressing said resin covering iron powder into iron powder or the iron powder which gave the phosphoric acid compound coat to the front face.

[Claim 9] It dries, after mixing the liquid which dissolved which resin powder for association of (1) - (6)

according to claim 7 in the organic solvent at iron powder or the iron powder which gave the phosphoric acid compound coat to the front face. A resin content with a mass ratio The manufacture approach of the dust core which considers as the resin covering iron powder which is below 0.3 mass %, and is characterized by heat-treating while adding any of said resin powder for association they are, making the whole quantity of said resin into 0.15 to 1 mass % with a mass ratio and pressing this mixed powder into said resin covering iron powder.

[Claim 10] The manufacture approach of a dust core given in any of claim [powder / said / for association / resin] 7 using the grain size of 1-150 micrometers to claim 9 they are.

[Claim 11] Compression molding of said mixed powder or resin covering iron powder is the manufacture approach of the dust core given in any of claims 7-10 they are performed in the condition of having heated to the temperature which said resin for association does not fuse.

[Claim 12] Compression molding of said mixed powder or resin covering iron powder is the manufacture approach of a dust core given in any of claims 7-10 which perform secondary compression molding in the condition of having heated to the temperature which said resin for association does not fuse after performing primary compression molding in the condition of not heating they are.

[Claim 13] Compression molding of said mixed powder or resin covering iron powder is the manufacture approach of the dust core given in any of claims 7-10 they are performed in the condition of having heated to the temperature which said resin for association softens or fuses.

[Claim 14] said compression -- the manufacture approach of a dust core given in any of claims 7-13 heated at the temperature which sets heat-treatment of an adult they to be [any under atmospheric air, inert gas, and reduced pressure], and at least one sort of said resin for association fuses.

[Claim 15] The manufacture approach of a dust core given in any of claims 11-13 heat-treated at the temperature which performs compression molding in the condition of having heated, sets the hot green compact released from mold they to be [any under atmospheric air, inert gas, and reduced pressure], and at least one sort of said resin for association fuses.

[Claim 16] The manufacture approach of a dust core given in any of claims 7-15 which perform stabilizing treatment which heats the dust core which heat-treated said compression-molding object at the temperature of 150-320 degrees C they are.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] Especially this invention relates to the dust core excellent in the metal mold moldability and the soft magnetism property, and its manufacture approach.

[0002]

[Description of the Prior Art] In connection with a miniaturization and densification of the electrical and electric equipment in recent years and electronic equipment, the demand of a RF iron core which possesses small and high flux density, permeability, and low iron loss also to the core ingredient used for these is increasing. As such a core ingredient, although the ferrite core is used, there is a fault that saturation magnetic flux density is low. On the other hand, in the dust core which combined alloy powder, such as Sendust and a permalloy, by insulating resin, such as phenol resin and an epoxy resin, although at least 100kHz or more of eddy current losses can be suppressed, the flux density of practical use is difficult to fill the demand of a miniaturization with extent higher than a ferrite core enough. Moreover, it is known as a motor iron core or an object for tolan scores that the dust core which made the magnetic particle the iron powder of a high grade has comparatively high flux density. this -- for example, the soft magnetism composite material (name: Soft Magnetic Composite:CMC) of HEGANESU -- it is -- the front face of the atomization iron powder of a high grade, or reduced iron powder -- the pole of a phosphoric acid system -- a thin insulating coat is formed, and thermosetting phenol resin and thermoplastic polyamide resin (nylon) are used, and it characterizes the resin for association by high flux density, high permeability, and low iron loss.

[0003]

[Problem(s) to be Solved by the Invention] Since a dust core which was described above has low reinforcement with mechanical ** which it is a low price and is promising for the miniaturization of a core, if it is easy to produce a crack and a deficit and temperature uses it under a high environment when performing cutting and drill drilling processing, a mechanical strength will tend to fall remarkably. That is, in the application of an automobile, an industrial machine, etc., this kind of soft magnetic materials have neither a fall on the strength nor a dimensional change while they are used in the environment where temperature is high, in many cases and maintain high magnetic properties in such an environment, and it has the material strength which can be equal to cutting etc., and moreover more much more price reduction is demanded. Of course, the frequency used is comparatively high and to be high permeability is also desired.

[0004]

[Means for Solving the Problem] As a result of canceling the technical problem which a dust core has conventionally [above-mentioned] how or coming examination in piles, by devising selection, an addition, resin powder, a mixed mode, etc. of the resin for association, this invention persons resulted in the corroboration that the above-mentioned technical problem is cancelable, and completed this invention.

[0005] In the dust core which combined iron powder or the iron powder which gave the phosphoric acid compound coat to the front face by resin, said resin for association is in following any, and invention of claims 1-4 is characterized by a resin content being 0.15 to 1 mass % of total mass.

(Claim 1) Polyphenylene sulfide (it is hereafter described as PPS), or thermoplastic polyimide (it is hereafter described as Thermoplasticity PI)

(Claim 2) The mixture (claim 4) PPS with the resin with glass transition higher than the mixture or Thermoplasticity PI, and said thermoplasticity PI with high resin of glass transition temperature from the

mixture (claim 3) PPS of PPS and Thermoplasticity PI and said PPS, Thermoplasticity PI, and glass transition temperature are mixture [0006] with resin [at least] higher than said PPS. Claim 5 specifies resin with a high glass transition temperature in said configuration. That is, as resin with a glass transition temperature higher than Thermoplasticity PI, it is in any of non-thermoplasticity polyimide, polyamidoimide, and polyamide bismaleimide. As resin with a glass transition temperature higher than PPS, it is in any of the resin mentioned above as what has a glass transition temperature higher than polyphenylene oxide, Pori Sall John, polyether sulfone, polyarylate, polyether imide, and said thermoplastic polyimide. Claim 6 makes the content of resin with a glass transition temperature higher than said PPS and said thermoplastic polyimide below one half in claims 3-5 among the total content 0.15 of the resin for association - 1 mass %. The central value of the glass transition temperature (a differential heating value, DSC:Differential ScanningCalorimetry) of each above resin was shown in Table 1.

[0007]

[Table 1]

樹脂名	ガラス転移温度 (℃)
PPS	90
熱可塑性PI	250
ポリアミドイミド	280
ポリアミノビスマレイミド	270
非熱可塑性ポリイミド	260
ポリエーテルスルフォン	225
ポリエーテルイミド	217
ポリフェニレンオキシサイド	210
ポリアリレート	193
ポリサルフォン	190

[0008] Claims 7-9 are the processes of the above dust core, and consist of the following configuration descriptions.

(Claim 7) While doing 0.15-1 mass % mixing of the powder of which (it mentioned to claims 1-4) the above mentioned resin for association with a mass ratio and pressing this mixed powder into iron powder or the iron powder which gave the phosphoric acid compound coat to the front face, it heat-treats.

(Claim 8) While it dries after mixing the liquid which fused which (it mentioned to claims 1-4) the above mentioned resin for association to the organic solvent, and considering as the resin covering iron powder whose content of the resin for association is 0.15 to 1 mass % in a mass ratio and pressing this resin covering iron powder into iron powder or the iron powder which gave the phosphoric acid compound coat to the front face, it heat-treats.

(Claim 9) In the above manufacture approach, it dries, after mixing the liquid which dissolved which (it mentioned to claims 1-4) the above mentioned resin for association in the organic solvent as the 1st step, and the content of said resin considers as the resin covering iron powder which is below 0.3 mass % with a mass ratio. It heat-treats, while adding any of said resin powder for association they are, making the whole quantity of said resin into 0.15 - 1% with a mass ratio and pressing this mixed powder into said resin covering iron powder as the 2nd step.

[0009] (Claims 10 and 11) In the above manufacture approach, although ***** [compression molding of that said resin powder for association has a desirable grain size of 1-150 micrometers said mixed powder, or resin covering powder] under ordinary temperature, it checks that it can carry out in the condition of having heated to the temperature which said resin for association does not fuse.

[0010] Moreover, they are details configurations, such as said heat-treatment conditions, after claim 11. That is, it specified that you could be the mode which heats the green compact fabricated in claims 11-13 without heating to the mode heated to the 1st as a gestalt of compression molding below at the temperature which does not carry out resin fusion, and the 2nd below at the temperature which does not carry out resin fusion, the mode

heated to the 3rd under the temperature which carries out resin fusion, and any. In claim 14, about heat-treatment of said compression-molding object, it set they to be [any under atmospheric air, inert gas, and reduced pressure], and the mode heated at the temperature which at least one sort of the resin for association fuses was specified. This is heating at 300- and 450 degrees C in 250-400 degrees C and Thermoplasticity PI in PPS. In claim 15, it specified performing compression molding in the condition of having heated, setting the hot green compact released from mold they being [any under atmospheric air, inert gas, and reduced pressure], and performing continuously the mode heat-treated at the temperature which at least one sort of said resin for association fuses, i.e., heating compacting, and heat-treatment of a green compact. In claim 16, the mode which heats the dust core which heat-treated the compression-molding object at the temperature of 150-320 degrees C, and performs stabilizing treatment was specified.

[0011]

[Embodiment of the Invention] Next, the above invention is clarified according to the gestalt and example of operation. In this explanation, mixing of the magnetic powder for invention, the resin for association and its content, magnetic powder, and resin, compression molding, heat-treatment, and stabilizing treatment are explained in full detail in order first. Then, an experimental typical thing is mentioned as an example and an advantage is clarified.

[0012] (1. Magnetic powder) Target iron powder can apply the iron powder by various processes, such as the atomizing method and reduction. The grain size of iron powder is chosen by the flux density demanded and the frequency domain used. Although particle diameter can use 200 micrometers or less generally used by powder metallurgy, when compressibility is taken into consideration, it is 150 micrometers or less. Since overcurrent loss becomes small and a RF property improves so that the particle diameter of iron powder is small, as for particle diameter, it is more desirable to be referred to as 100 micrometers or less. Although it is not necessary to limit especially about a fine particle, since powder compressibility and powder flowability worsen and a high-density dust core is not acquired, as for the particle size distribution with many fine particles, it is desirable to consider as powder with a particle diameter of 10 micrometers or more.

[0013] The coat acts as an insulating layer, the iron powder which gave the phosphoric acid compound coat to the front face has the effectiveness which controls overcurrent generating between iron powder children, by existence of the resin for association, the effectiveness which controls generating of an overcurrent becomes still higher, and its RF property improves more. Phosphoric acid iron, phosphoric acid manganese, zinc phosphate, calcium phosphate, etc. are suitable for the phosphoric acid compound for coats. Moreover, the commercial item of the iron powder which gave the phosphoric acid acid compound coat to the front face does not interfere, either. As this example, the iron powder made from HEGANESU (trade name-ermite or Somaloy) etc. is mentioned.

[0014] (2. Joint resin) As resin for association, PPS which is excellent in thermal resistance, and Thermoplasticity PI show [a good property] and are suitable. It is the environment where the temperature for which a dust core is used exceeds 180 degrees C, and when used over long duration, aging is produced in the configuration of a dust core, and a dimension, or a possibility that the apparent insulating engine performance may fall is in them. It being thought that the reason has the complicated residual stress's which produces the former's at the time of compression molding, and the latter can consider possibility that the thickness of the insulating resin between magnetic powder will decrease, according to hot environments. A property will be improved by such fear if resin with a glass transition point higher than these is mixed to said PPS or Thermoplasticity PI. Since the resin between magnetic particles (iron grain child) is in the compound condition that heat characteristics differ, this is considered with making deformation in use and migration hard to produce. Let the content of resin with a high glass transition point be the range which does not exceed PPS which serves as a subject, or the amount of Thermoplasticity PI. It is also the same thing as this technical thought to mix Thermoplasticity PI to PPS. [0015] (3. Content of joint resin) The range of 0.15 - 1 mass % of total mass is suitable for the content of the resin for association. Under by 0.15 mass %, there is little effectiveness which the particle of magnetic powder combines and insulates, and it becomes inadequate [the reinforcement of a dust core], and the insulation between magnetic powder will become bad. Moreover, since the pulse duty factor of the magnetic particle occupied to a dust core will become low and will become what has the low consistency of a magnetic particle while the reinforcement of a dust core and insulation will become high if the content of the resin for association exceeds 1 mass %, high flux density and high permeability are no longer obtained.

[0016] In relation with permeability, the thing which has many contents of resin becomes low in an about 50Hz low frequency field. In a field with a high frequency of about 5kHz, a value far lower than the permeability in a low frequency field is shown, and, as for what does not contain resin, a resin content serves as [permeability] the almost same value as permeability [in / maximum is shown and / low frequency] near the 0.3 mass % compared with this. If a resin content increases further, it falls gradually like the permeability in the case of low frequency, and if a resin content exceeds 1 mass %, it will become lower than permeability in case resin is not included. As for the content of the resin for association, 0.15 - 1 mass % becomes the optimal also from the relation of such the amount of resin and permeability. Especially a resin content is desirable near the 0.3 mass %. Moreover, consistencies are 7.35 g/cm³. It is desirable that it is above.

[0017] (4. Mixing of magnetic powder and the resin for association) The resin for association insulates between magnetic powder children, and controls generating of an overcurrent. Although the iron powder which gave the phosphoric acid compound coat has a possibility that the insulation with a phosphoric acid compound may be broken according to exfoliation or omission in the case of powder compression molding, it is protected by existence of the resin for association, and can control generating of an overcurrent more.

[0018] The resin for association is mixable in a powdered form. If it is the particle size distribution, EQC, or thin jar of magnetic powder in that case, the mixed state will become good and thermal resistance's will improve. In order to raise the insulation between magnetic powder, it is desirable to consider as the grain size of 60 micrometers or less. Moreover, after making the resin for association add and hypoviscosity-ize an organic solvent with strong polarities, such as an n-methyl-2-pyrrolidone, and performing coating of a complement to iron powder using the coating equipment of a fluid bed type or a churning mixing type, the approach of drying is also desirable.

[0019] Moreover, it is good also as an approach of drying it, after coating the resin for association containing an organic solvent with the amount of resin smaller than the case where it describes above, and making resin covering powder, and mixing the powder of the resin for association to this resin covering powder. The resin coat obtained using the resin for association containing an organic solvent becomes that in which the insulating engine performance was more excellent. Generating of an overcurrent of thickness of resin decreases that it is 20nm or more. In order to obtain the resin coat of 20nm of thickness, the resin solution which serves as amount of resin 0.15 mass % extent about is mixed. On the other hand, if resin thickness exceeds 200nm, powdered compressibility will worsen, consequently it will become the dust core where magnetic properties are inadequate. If the resin powder for association is mixed by addition to the magnetic powder which covered resin, covering resin will be protected, and the more excellent magnetic property is acquired.

[0020] (5. Compression molding) Compression molding of the resin covering magnetism powder is carried out using metal mold. It is desirable at the time of compression molding to apply beforehand shaping lubricant powder usually used for a metal mold side by powder metallurgy for reduction of compressive improvement or green compact draw friction, such as zinc stearate and ethylene bis-steer ROAMAIDO, with a electrostatic-spraying cloth etc. Moreover, in order to fabricate in a higher consistency, after pressing the 1st order in the condition do not heat neither the mode and the mixed powder which perform in the condition heated to the temperature which the resin for association does not fuse, nor resin coat iron powder, it is carrying out in the mode which performs secondary compression molding in the condition having heated to the temperature which the resin for association does not fuse, and the mode which perform compression formation in the condition having heated to the temperature which fuses from the temperature which the resin for association softens further. In addition, as shaping after treatment, after fabricating, it is good also as an approach of performing heat-treatment which cools to ordinary temperature and is described henceforth, but since the approach, then heat energy which shift to heat-treatment while the Plastic solid has been hot, and a cooldown delay can be excluded after fabricating, it is rational.

[0021] (6. Heat-treatment) Heat-treatment is a process which is made to carry out melting of the resin for association, and attains stabilization of the resin property by crystallization of the resin for association further. Heating time is selected by the class of resin to be used whenever [stoving temperature]. Temperature is range where resin does not heat-deteriorate from the melting point of resin, and is made into 300-450 degrees C by 250-400 degrees C and Thermoplasticity PI at PPS. Generally heating time is about about 0.5 - 1 hour.

[0022] The ambient atmosphere at the time of heating can be performed in atmospheric air. However, existence of the oxygen in atmospheric air can consider a possibility of producing the fall of resin on the strength, and the

fall of a mechanical property. This is because it may also happen for the polymerization reaction of resin to advance, to become easy to generate a gas condensate, and to remain as air bubbles in resin by existence of oxygen. Therefore, in advance of heating in atmospheric air, it is more preferably heated in inert gas ambient atmospheres, such as nitrogen gas. Moreover, if it heats in the decompressed ambient atmosphere, while the amount of oxygen of an ambient atmosphere will decrease, a gas condensate can be made to emit from resin more. These ambient atmospheres are combinable suitably. In the cooling process of heat-treatment, if it cools over many hours in a field with a temperature of about 320-150 degrees C, it can serve as the stabilizing treatment described below.

[0023] (7. Stabilizing treatment) When stabilizing treatment was performed, the property of the resin for association is stabilized and a dust core is used at high temperature, it shall be hard to produce aging. In this case, said heat-treatment is performed, and after cooling, it is once heated at about 150-320 degrees C for about 1 to 2 hours. Moreover, it can be based on the approach of holding in an about 320-150-degree C temperature field for 1 to 2 hours by the cooling process of said heat-treatment.

[0024]

[Example] Next, an invention configuration and an advantage are clarified by the example and the example of a comparison of this invention.

(1) The prepared powder is eight kinds of following (1) - (8).

(1) . atomization iron powder: -- "product [made from HEGANESU] and lot number:ABC 100.30" grain size of this is a thing 150 micrometers or less (it is hereafter described as pure iron powder).

(2) . phosphoric acid coat processing atomization iron powder: -- "product [made from HEGANESU] and lot number:Somaloy500" grain size of this is a thing 150 micrometers or less (it is hereafter described as coat formation iron powder).

(3) -- phosphoric acid coat processing atomization iron powder containing . thermoplasticity polyamide resin (it is hereafter described as polyamide): -- this does 0.6 mass % mixing of a thermoplastic polyamide at "marketing powder made from HEGANESU" phosphoric acid coat processing atomization iron powder (Somaloy500).

(4) .PPS powder: -- the "Dainippon Ink grain size" of this is a thing 150 micrometers [or less (-150 micrometers)] and a thing 60 micrometers or less (-60 micrometers) (it is hereafter described as PPS).

(5) . thermoplasticity polyimide powder: -- the "Mitsui Chemicals make" and grain size of this are things (a thing 150 micrometers or less (-150 micrometers) and 60 micrometers or less (-60 micrometers)) (it is hereafter described as Thermoplasticity PI).

(6) . thermosetting polyimide powder: -- "the product made from low RANNU" and grain size of this are things 150 micrometers or less (-150 micrometers) (it is hereafter described as Thermosetting PI).

(7) . thermosetting phenol resin powder: -- the "Dainippon Ink make" and grain size of this are things 150 micrometers or less (-150 micrometers) (it is hereafter described as a phenol).

(8) . zinc stearate powder: -- this is lubricant for dice currently generally used.

[0025] (2)

[Example 1] specified quantity (by all mass %, it becomes 0%, 0.15%, 0.3%, 4.5%, 6.0%, 7.5%, 1.0%, and 1.2% -- as) mixing of PPS (-150 micrometers) was carried out at the effective-permeability aforementioned coat processing iron powder of the content of resin, and a dust core, and mixed powder was pressed into the ring configuration ($\phi 10 \times \phi 23 \times 5 \text{mm}$) by compacting pressure 1470MPa using the metal mold which applied zinc stearate powder. After heating a Plastic solid at the temperature of 320 degrees C in air for 1 hour, it acquired the dust core by heating for 1 hour and cooling at the temperature of 240 degrees C. In addition, it is the pure green compact which does not contain PPS.

[0026] The effective permeability was measured with the B-H analyzer. Frequencies are 50Hz and 5000Hz, and impression flux density is 1T (Tess Ra). The measurement result of the effective permeability is as drawing 1 . That is, the effective permeability in 50Hz is falling almost linearly to the increment in a resin content. On the other hand, that in which the effective permeability in 5000Hz does not contain PPS is low, and an PPS content serves as maximum near the 0.3 mass %, and with the PPS content beyond it, it is falling gently, and when an PPS content is 1 mass %, it is almost the same as the value of the dust core which does not contain PPS. In addition, also in the case of other resin powder, it becomes the same inclination although resin gave the example of PPS.

[0027] About the resin content of invention, in the field with few resin contents, since the average rate of

change of the effective permeability were large, from the above thing, the resin content was carried out to more than 0.15 mass %. In the side with many resin contents, it considered as the resin content below 1.0 mass % which does not become lower than the effective permeability of the dust core which does not contain resin.

[0028] (3)

[Example 2] Specified quantity addition of each resin powder was carried out at the heat-resistant aforementioned pure iron powder of the class of resin, and a dust core using pure iron powder, and it mixed by the V shaped rotary mixer. The mixed rate of resin powder is as being shown in Table 2. PPS and Thermoplasticity PI used that whose grain size is -150 micrometers.

[Table 2]

試料 No.	樹脂粉末の種類と含有量 (質量%)				備考
	PPS	熱可塑性PI	熱硬化性PI	フェノール	
A 1	0.15				発明
A 2	0.6				"
A 3	1.0				"
A 4		0.6			"
A 5	0.3		0.3		"
A 6		0.3	0.3		"
A 7				0.6	比較例

[0029] Each mixed powder was pressed by compacting pressure 1470MPa a cylinder ($\phi 23 \times 5$ mm) and in the shape of a cylindrical shape ($\phi 10 \times \phi 23 \times 10$ mm). shaping -- the internal surface of metal mold -- beforehand -- zinc stearate -- a electrostatic-spraying cloth -- carrying out -- mixed powder -- restoration -- and powder compacting was carried out. That in which that in which the heat-treatment temperature of a Plastic solid includes the thing in which resin contains PPS, and Thermoplasticity PI contains 320 degrees C and a phenol considered as 150 degrees C, and was heated in nitrogen-gas-atmosphere mind for 1 hour, respectively. Stabilizing treatment was performed about that in which resin includes PPS and Thermoplasticity PI, and was heated at 240 degrees C in atmospheric air for 1 hour.

[0030] Cutting of each cylindrical shape-like ($\phi 23 \times 5$ mm) sample was carried out to the prism configuration of $5 \times 23 \times 5$ mm, and the insulating performance evaluation was presented with it. an insulating performance evaluation -- constant temperature with a temperature of 200 degrees C -- after heating in a layer for 100 hours, the decreasing rate to the value before measuring and heating the appearance specific resistance value by the four probe method (sample both ends are touched in a direct current, two terminals are touched a sink and between them, and electric resistance is measured) estimated. Moreover, the radial-crushing-strength value was measured about each cylindrical shape-like ($\phi 10 \times \phi 23 \times 10$ mm) sample. Radial crushing strength is maximum load until it compresses and destroys by per minute 0.5mm of compression velocity in a room temperature and the temperature of 200 degrees C. It saw in Table 3 and the measurement result with specific resistance, a room temperature, and a radial crushing strength of 200 degrees C was shown.

[Table 3]

試料 No.	見掛け固有抵抗 ($m\Omega \cdot cm$)			圧縮強度 (MPa)			備考
	初期値	加熱後	低下率 (%)	室温	200℃	低下率 (%)	
A 1	2. 0	1. 1	45	185	150	19	発明
A 2	5. 0	3. 5	30	110	80	27	"
A 3	7. 0	5. 7	19	75	50	33	"
A 4	5. 0	3. 5	30	110	90	18	"
A 5	5. 0	4. 3	14	110	80	27	"
A 6	5. 0	4. 5	10	110	85	23	"
A 7	7. 0	1. 2	83	90	30	67	比較例

[0031] (Evaluation) Appearance specific resistance rises linearly by the increment in the content of resin. Although a resin content sees in 0.15 mass % and 1 mass % and specific resistance differs, it is usable with the application of a dust core, respectively. If the appearance specific resistance before and behind heating by the class of resin is seen, even if the thing (a sample A1 - A3) containing PPS changes a resin content, its variation before and behind heating is almost the same, and it has decreased in the decreasing rate (rate of change), so that there are many resin contents. A thing (sample A4) including Thermoplasticity PI is also that it is the same. The variation of the thing [the thing (sample A5) which includes Thermosetting PI in PPS, and] (sample A6) which includes Thermosetting PI in Thermoplasticity PI of appearance specific resistance has become less than the thing of PPS. Although the appearance specific resistance before heating is higher than the thing containing PPS etc., what contains a phenol compared with these (sample A7) has very much variation after heating, and is what has it. [remarkable the appearance specific resistance after heating and low]

[0032] Radial crushing strength becomes so low that there are many resin contents. a thing (sample A4) including the thing (a sample A1 - A3) in which a difference (a room temperature and 200 degrees C) contains PPS, and Thermoplasticity PI -- although the reinforcement of a room temperature of what contains a phenol although it is almost [both] the same (sample A7) is also low, it is what has it. [remarkable the reinforcement in 200 degrees C and low]

[0033] (4)

[Example 3] The mixed powder which carried out specified quantity addition of each resin shown in Table 4 at the heat-resistant aforementioned coat formation iron powder of the class of resin using coat formation iron powder and a dust core was produced. In addition, the polyamide of a sample B16 is the commercial powder carried out 0.6 mass % mixing about a polyamide at the above-mentioned phosphoric acid coat processing atomization iron powder (Somaloy500). After using a sample B13 as the magnetic powder with which it dried and by which the liquid which added the n-methyl-2-pyrrolidone as an organic solvent to PPS was added to coat formation iron powder, and it mixed, and the PPS content was covered by 0.15 mass %, it is the mixed powder which mixed PPS further and made the PPS content 0.6 mass %. The other mixed powder added the powder of resin to coat formation iron powder, and mixed it by the V shaped rotary mixer. Sample B14-16 are an example of a comparison. Each mixed powder was pressed on the same conditions as said example 2 a cylinder (phi23x5mm) and in the shape of a cylindrical shape (phi10xphi23x10mm).

[0034] That in which that in which that in which that in which, as for the heat-treatment temperature of a Plastic solid, resin includes PPS and Thermoplasticity PI includes 320 degrees C and Thermosetting PI contains 200 degrees C and a phenol contains 150 degrees C and a polyamide considered as 275 degrees C, and was heated in nitrogen-gas-atmosphere mind for 1 hour, respectively. In addition, about the sample B12, it carried out in air. Stabilizing treatment was performed about that in which resin includes PPS and Thermoplasticity PI, and was heated at the temperature of 240 degrees C for 1 hour.

[0035]

[Table 4]

試料 No.	樹脂の種類と含有量 (質量%)						
	P P S -150	P P S -60	熱可塑性 PI -150	熱可塑性 PI -60	熱硬化性 PI	フェノ ール	ポリ アミド
B 1	0. 1 5						
B 2	0. 3						
B 3	0. 6						
B 4	1. 0						
B 5	0. 1		0. 0 5				
B 6	0. 3		0. 3				
B 7			0. 6				
B 8	0. 3				0. 3		
B 9			0. 3		0. 3		
B10		0. 6					
B11				0. 6			
B12	0. 6						
B13	0. 6						
B14					0. 6		
B15						0. 6	
B16							0. 6

[0036] By the same approach as said example 2, the radial crushing strength in an appearance specific resistance value, and a room temperature and temperature of 200 degrees C after heating at the temperature of 200 degrees C for 100 hours was measured. A measurement result is as that of Table 5.

[0037]

[Table 5]

試料 No.	見掛け固有抵抗($m\Omega \cdot cm$)			圧壊強度(MPa)			備考
	初期値	加熱後	低下率(%)	室温	200℃	低下率(%)	
B 1	5.0	3.5	30	185	150	19	発明
B 2	8.0	6.0	25	155	120	23	〃
B 3	12.0	10.0	17	110	80	27	〃
B 4	17.5	16.0	9	75	50	33	〃
B 5	5.5	3.5	30	185	150	19	〃
B 6	12.0	10.0	17	110	80	27	〃
B 7	12.0	10.0	17	110	95	14	〃
B 8	12.0	11.0	8	110	80	27	〃
B 9	12.0	11.5	4	110	80	27	〃
B10	15.0	13.5	13	130	100	23	〃
B11	15.0	13.0	10	110	90	18	〃
B12	17.0	13.0	24	110	80	27	〃
B13	13.0	12.0	8	110	85	23	〃
B14	9.0	8.0	11	90	70	22	比較例
B15	10.0	4.0	60	90	30	67	〃
B16	8.0	3.0	63	90	20	78	〃

[0038] (Evaluation) Appearance specific resistance is higher than the dust core which used pure iron powder. It turns out that the insulation of an iron powder child is good as there is a phosphoric acid compound coat. It is the same as that of the thing of pure iron powder to go up linearly by the increment in the content of resin. If the appearance specific resistance before and behind heating by the class of resin is seen, the thing containing PPS has almost the same variation irrespective of a resin content, and in a decreasing rate (rate of change), it will decrease, so that there are many resin contents. Moreover, although the thing of mixing with the thermoplasticity PI the thing of mixture with PPS whose grain size is -150 micrometers, this PPS and Thermoplasticity PI, or Thermosetting PI, and whose grain size are -150 micrometers, and this Thermoplasticity PI and Thermosetting PI shows the almost same property, there are few falls of the appearance specific resistance according [that to which Thermosetting PI is mixed with PPS or Thermoplasticity PI in details] to heating.

[0039] What set to -60 micrometers grain size of the powder which added PPS and Thermoplasticity PI is higher [heating order] than a thing with a grain size of -150 micrometers. What carried out wet blending of PPS of a sample B13, covered it, and mixed PPS is seen slightly, and its specific resistance is higher than what was mixed with powder. Although what heat-treated the sample B13 in air has the large fall of the appearance specific resistance by heating, the value higher than heating among nitrogen gas is shown. Compared with these, the thing (sample B15) containing a phenol and the thing (sample B16) of a polyamide have low initial value, and the heated amount of falls is large. Moreover, by the thing (sample B16) including Thermosetting PI, although there are few amounts of falls by heating, the low value is shown.

[0040] Radial crushing strength is the almost same level as the case of pure iron powder, and shows the inclination for the amount of falls when heating at relation with a resin content and 200 degrees C to be the same. In radial crushing strength, a difference is not accepted in the difference in the effect of the grain size of resin powder, wet covering of resin, and the ambient atmosphere of heat-treatment. Although the amount of falls by heating has little that the thing containing a phenol and the thing of a polyamide have low initial value compared with an PPS system and thermoplastic PI system, and the amount of falls when heating is large, and thermosetting PI, it is the same as the rank in the case of appearance specific resistance that the low value is shown.

[0041]

[Effect of the Invention] As explained above, the dust core of this invention is the form of the mixture of PPS resin or thermoplastic PI resin, and these and resin with a comparatively high glass transition temperature at iron powder or the iron powder which gave the phosphoric acid compound coat to the front face. By 0.15-1 mass % Containing, permeability is high and the property which was excellent when used especially in a RF field is shown. Moreover, even when used in the environment where temperature is high, since specific resistance and strength in high temperature are high, it can contribute to the engine performance of the equipment used, and a miniaturization, and the applicability of a dust core can be expanded.

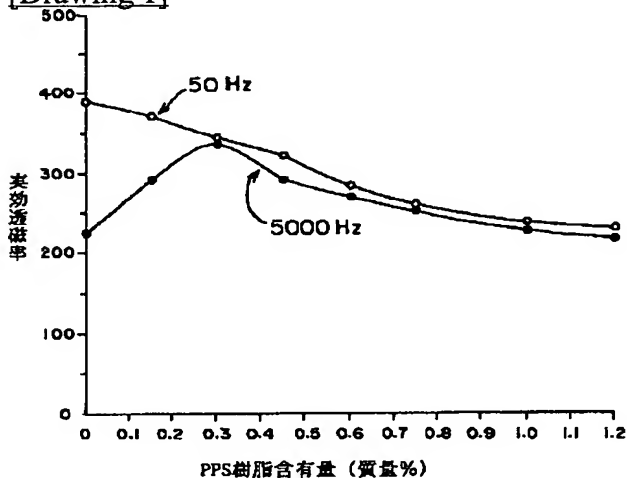
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DRAWINGS

[Drawing 1]

[Translation done.]

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(54) DUST CORE AND ITS MANUFACTURING METHOD

(57)Abstract:

PROBLEM TO BE SOLVED: To realize such a dust core that can keep a magnetic characteristic even when it is used in a high-temperature environment and shows no reduction of strength or no dimensional change, and can endure cutting or the like.

SOLUTION: This dust core is formed by joining an iron powder or an iron powder whose surface is covered with a phosphate compound film, with a resin. The joint resin is made of polyphenylene sulfide or thermoplastic polyimide, and the content of resin is 0.15 to 1 mass percentage of total mass.

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[Claim 1] A dust core comprising iron powders, or iron powders having a surface coated with a phosphoric acid compound, said iron powders being bound with a resin, wherein the binding resin is polyphenylene sulfide or thermoplastic polyimide, and a content of a resin is 0.15 to 1% by mass of total mass.

[0020] (5. Compression molding) A resin-coated magnetic powder is compression-molding using a mold. Upon compression molding, it is desirable to coat a molding lubricant powder such as zinc stearate and ethylene bisstearoamide on a mold surface by electrostatic coating in advance, in order to improve compressibility or reducing green compact ejection friction. In addition, in order to mold to a higher density, there are an aspect of performing molding in the state where heated to a temperature at which a binding resin is not melted, an aspect of performing primary compression molding in the state where a mixed powder or a resin-coated iron powder is not heated, and, thereafter, performing secondary compression molding in the state where heated to a temperature at which a binding resin is not melted, and an aspect of performing compression molding in the state where heated from a temperature at which a binding resin is softened, to a temperature at which a binding resin is melted. In addition, as molding posttreatment, there may be a method of molding and, thereafter, cooling a molded article to a normal temperature, and performing heating treatment described below, or a method of performing molding and, thereafter, transferring to heating treatment while a molded article is hot, and thermal energy and cooling time can be omitted,

being reasonable.

[0025](2)

[Example 1] Content of resin and effective permeability of dust core

A prescribed amount (in terms of total % by mass, 0 %, 0.15 %, 0.3 %, 4.5 %, 6.0 %, 7.5 %, 1.0 %, 1.2 %) of PPS (-150 μm) was mixed into the aforementioned coating-treated iron powder, and the mixed powder was compression-molded into a ring shape ($\phi 10 \times \phi 23 \times 5\text{mm}$) at a molding pressure of 1470MPa using a mold coated with a zinc stearate powder. A molded article was heated at a temperature of 320 °C for 1 hour in the air, heated at a temperature of 240 °C for 1 hour, and cooled, thereby, a dust core was obtained. What does not contain PPS is a pure green compact.

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(54) 【発明の名称】 圧粉磁心及びその製造方法

(57) 【要約】 (修正有)

【課題】 圧粉磁心として、高い温度環境における使用でも磁気特性を維持でき、強度低下や寸法変化がなく、切削加工等に耐えうるものを実現する。

【解決手段】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉を樹脂で結合した圧粉磁心を対象とする。構成特徴は、結合用樹脂がポリフェニレンサルファイド及び熱可塑性ポリイミドの何れかであり、樹脂含有量が全質量の0.15～1質量%となっている。

【特許請求の範囲】

【請求項1】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉を樹脂で結合した圧粉磁心において、前記結合用樹脂はポリフェニレンサルファイド及び熱可塑性ポリイミドの何れかであり、樹脂含有量が全質量の0.15～1質量%となっている、ことを特徴とする圧粉磁心。

【請求項2】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉を樹脂で結合した圧粉磁心において、前記結合用樹脂はポリフェニレンサルファイド及び熱可塑性ポリイミドの混合物であり、樹脂含有量が全質量の0.15～1質量%となっている、ことを特徴とする圧粉磁心。

【請求項3】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉を樹脂で結合した圧粉磁心において、前記結合用樹脂はポリフェニレンサルファイド（以下、PPSという）及び前記PPSよりガラス転移温度の高い樹脂との混合物、又は、熱可塑性ポリイミド及び前記熱可塑性ポリイミドよりガラス転移温度の高い樹脂との混合物の何れかであり、樹脂含有量が全質量の0.15～1質量%となっている、ことを特徴とする圧粉磁心。

【請求項4】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉を樹脂で結合した圧粉磁心において、前記結合用樹脂はポリフェニレンサルファイド（以下、PPSという）と熱可塑性ポリイミド及びガラス転移温度が少なくとも前記PPSより高い樹脂との混合物であり、樹脂含有量が全質量の0.15～1質量%となっている、ことを特徴とする圧粉磁心。

【請求項5】 前記熱可塑性ポリイミドよりガラス転移温度の高い樹脂が、非熱可塑性ポリイミド、ポリアミドイミド、ポリアミノビスマレイミドの何れかであり、前記PPSよりガラス転移温度の高い樹脂が、ポリフェニレンオキサイド、ポリサルフォン、ポリエーテルサルフォン、ポリアリレート、ポリエーテルイミド、及び前記熱可塑性ポリイミドよりガラス転移温度の高いものとして前記に挙げた樹脂の何れかである請求項3又は請求項4に記載の圧粉磁心。

【請求項6】 前記PPSよりガラス転移温度の高い樹脂又は前記熱可塑性ポリイミドよりガラス転移温度の高い樹脂の含有量が、結合用樹脂の全含有量0.15～1質量%のうち、半分以下である請求項3から5の何れかに記載の圧粉磁心。

【請求項7】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉に、下記（1）から（6）に記載の何れかの結合用樹脂粉末を質量比で0.15～1質量%混合し、この混合粉を圧縮成形すると共に加熱処理することを特徴とする圧粉磁心の製造方法。

（1）ポリフェニレンサルファイド（以下、PPSという）

（2）熱可塑性ポリイミド

（3）前記PPSと熱可塑性ポリイミドの混合物

（4）前記PPS及び前記PPSよりガラス転移温度の高い樹脂との混合物

（5）前記熱可塑性ポリイミド及び前記熱可塑性ポリイミドよりガラス転移温度の高い樹脂との混合物

（6）前記PPSと前記熱可塑性ポリイミド及びガラス転移温度が少なくとも前記PPSより高い樹脂との混合物

【請求項8】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉に、請求項7に記載の（1）～（6）の何れかの結合用樹脂粉末を有機溶剤に溶解した液を混合したのち乾燥して、前記樹脂含有量が質量比で0.15～1質量%である樹脂被覆鉄粉とし、前記樹脂被覆鉄粉を圧縮成形すると共に加熱処理することを特徴とする圧粉磁心の製造方法。

【請求項9】 鉄粉、又は磷酸化合物被膜を表面に施した鉄粉に、請求項7に記載の（1）～（6）の何れかの結合用樹脂粉末を有機溶剤に溶解した液を混合したのち乾燥して、樹脂含有量が質量比で、0.3質量%以下である樹脂被覆鉄粉とし、

前記樹脂被覆鉄粉に前記結合用樹脂粉末の何れかを添加して前記樹脂の全量を質量比で0.15～1質量%とし、この混合粉を圧縮成形すると共に加熱処理することを特徴とする圧粉磁心の製造方法。

【請求項10】 前記結合用樹脂粉末が粒度1～150μmものを用いる請求項7から請求項9の何れかに記載の圧粉磁心の製造方法。

【請求項11】 前記混合粉又は樹脂被覆鉄粉の圧縮成形は、前記結合用樹脂が溶融しない温度に加熱した状態で行う請求項7から10の何れかに記載の圧粉磁心の製造方法。

【請求項12】 前記混合粉又は樹脂被覆鉄粉の圧縮成形は、加熱しない状態で1次圧縮成形を行ったのち、前記結合用樹脂が溶融しない温度に加熱した状態で2次圧縮成形を行う請求項7から10の何れかに記載の圧粉磁心の製造方法。

【請求項13】 前記混合粉又は樹脂被覆鉄粉の圧縮成形は、前記結合用樹脂が軟化又は溶融する温度に加熱した状態で行う請求項7から10の何れかに記載の圧粉磁心の製造方法。

【請求項14】 前記圧縮成体の加熱処理は、大気中、不活性ガス中、減圧中の何れかにおいて、前記結合用樹脂の少なくとも1種が溶融する温度で加熱する請求項7から13の何れかに記載の圧粉磁心の製造方法。

【請求項15】 加熱した状態で圧縮成形を行い、離型された熱い圧粉体を大気中、不活性ガス中、減圧中の何れかにおいて、前記結合用樹脂の少なくとも1種が溶融する温度で加熱処理する請求項11から13の何れかに記載の圧粉磁心の製造方法。

【請求項16】 前記圧縮成形体を加熱処理した圧粉磁

心を、温度150～320℃で加熱する安定化熱処理を施す請求項7から15の何れかに記載の圧粉磁心の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、特に、金型成形性及び軟磁性特性に優れた圧粉磁心及びその製造方法に関する。

【0002】

【従来の技術】近年の電気、電子機器の小型化及び高密度化に伴い、これらに使用される磁心材料に対しても小型で高い磁束密度と透磁率及び低鉄損を具備する高周波鉄心の要求が高まっている。このような磁心材料としては、フェライトコアが使用されているものの飽和磁束密度が低いという欠点がある。これに対し、センダスト、バーマロイ等の合金粉末をフェノール樹脂やエポキシ樹脂等の絶縁性樹脂で結合した圧粉磁心においては、100kHz以上でも渦電流損失を抑えられるが、実用の磁束密度はフェライトコアより高い程度で、小型化の要求を充分満たすことは困難である。また、モータ鉄心やトランスコア用として、磁性粒子を高純度の鉄粉とした圧粉磁心は、比較的高い磁束密度を有することが知られている。これは例えば、ヘガネス社の軟磁性複合材料（名称：Soft Magnetic Composite:CMC）であり、高純度のアトマイズ鉄粉や還元鉄粉の表面に燐酸系の極薄い絶縁被膜を形成し、結合用樹脂を熱硬化性フェノール樹脂や熱可塑性ポリアミド樹脂（ナイロン）を使用するもので、高磁束密度、高透磁率、低鉄損が特徴とされる。

【0003】

【発明が解決しようとする課題】上記したような圧粉磁心は、低価格であり、コアの小形化に有望なものであるが、機械的な強度が低いため、切削加工やドリル孔開け加工を行う場合に割れや欠損を生じやすく、また、温度が高い環境の下で使用すると機械的強度が著しく低下しやすい。即ち、この種の軟磁性材料は、自動車や産業機械等の用途において、温度の高い環境で使用されることが多く、そのような環境で高い磁気特性を維持すると共に強度低下や寸法変化がなく、切削加工等に耐える材料強度を持ち、しかもより一層の価格低減が要求されている。勿論、用いられる周波数は比較的高いものとなっており、高透磁率であることも望まれる。

【0004】

【課題を解決するための手段】本発明者らは、上記従来圧粉磁心の持つ課題を如何に解消するか検討を重ねてきた結果、結合用樹脂の選定、添加量、樹脂粉末や混合態様等を工夫することにより上記課題を解消できるとの確証に至り、本発明を完成した。

【0005】請求項1～4の発明は、鉄粉、又は燐酸化合物被膜を表面に施した鉄粉を樹脂で結合した圧粉磁心において、前記結合用樹脂が下記の何れかであって、樹

脂含有量が全質量の0.15～1質量%であることを特徴としている。

（請求項1）ポリフェニレンサルファイド（以下、PPSと記す）、又は、熱可塑性ポリイミド（以下、熱可塑性PIと記す）

（請求項2）PPSと熱可塑性PIとの混合物

（請求項3）PPS及び前記PPSよりガラス転移温度の高い樹脂との混合物、又は、熱可塑性PI及び前記熱可塑性PIよりガラス転移の高い樹脂との混合物

（請求項4）PPSと熱可塑性PI及びガラス転移温度が少なくとも前記PPSより高い樹脂との混合物

【0006】請求項5は前記構成において、ガラス転移温度の高い樹脂を特定したものである。即ち、熱可塑性PIよりガラス転移温度の高い樹脂としては、非熱可塑性ポリイミド、ポリアミドイミド、ポリアミドビスマレイミドの何れかである。PPSよりガラス転移温度の高い樹脂としては、ポリフェニレンオキサライド、ポリサルフォン、ポリエーテルスルフォン、ポリアリレート、ポリエーテルイミド、及び前記熱可塑性ポリイミドよりガラス転移温度の高いものとして前記に挙げた樹脂の何れかである。請求項6は請求項3から5において、前記PPSや前記熱可塑性ポリイミドよりガラス転移温度の高い樹脂の含有量を、結合用樹脂の全含有量0.15～1質量%のうち、半分以下としたものである。以上の各樹脂のガラス転移温度（示差熱量、DSC：Differential Scanning Calorimetry）の代表値を表1に示した。

【0007】

【表1】

樹脂名	ガラス転移温度（℃）
PPS	90
熱可塑性PI	250
ポリアミドイミド	280
ポリアミノビスマレイミド	270
非熱可塑性ポリイミド	260
ポリエーテルスルフォン	225
ポリエーテルイミド	217
ポリフェニレンオキサライド	210
ポリアリレート	193
ポリサルフォン	190

【0008】請求項7～9は以上の圧粉磁心の製法で、次のような構成特徴からなる。

（請求項7）鉄粉、又は燐酸化合物被膜を表面に施した鉄粉に、前記した（請求項1～4に挙げた）何れかの結合用樹脂の粉末を質量比で0.15～1質量%混合し、この混合粉を圧縮成形すると共に加熱処理する。

（請求項8）鉄粉、又は燐酸化合物被膜を表面に施した鉄粉に、前記した（請求項1～4に挙げた）何れかの結

合用樹脂を有機溶剤に溶解した液を混合したのち乾燥して、結合用樹脂の含有量が質量比で0.15~1質量%である樹脂被覆鉄粉とし、この樹脂被覆鉄粉を圧縮成形すると共に加熱処理する。

(請求項9)以上の製造方法において、第1段階として、前記した(請求項1~4に挙げた)何れかの結合用樹脂を有機溶剤に溶解した液を混合したのち乾燥して、前記樹脂の含有量が質量比で、0.3質量%以下である樹脂被覆鉄粉とする。第2段階として、前記樹脂被覆鉄粉に前記結合用樹脂粉末の何れかを添加して前記樹脂の全量を質量比で0.15~1%とし、この混合粉を圧縮成形すると共に加熱処理する。

【0009】(請求項10と11)以上の製造方法において、前記結合用樹脂粉末は粒度1~150 μm ものが好ましいこと、前記混合粉又は樹脂被覆粉末の圧縮成形は常温下でもよいが、前記結合用樹脂が溶融しない温度に加熱した状態で行うことができることを確認したものである。

【0010】また、請求項11以降は前記加熱処理条件等の細部構成である。即ち、請求項11~13では、圧縮成形の形態として、第1に樹脂溶融しない温度以下で加熱する態様、第2に加熱しないで成形した圧粉体を樹脂溶融しない温度以下で加熱する態様、第3に樹脂溶融する温度下で加熱する態様、何れであってもよいことを特定した。請求項14では、前記圧縮成形体の加熱処理について、大気中、不活性ガス中、減圧中の何れかにおいて、結合用樹脂の少なくとも1種が溶融する温度で加熱する態様を特定した。これは例えば、PPSでは250~400 $^{\circ}\text{C}$ 、熱可塑性PIでは300~、450 $^{\circ}\text{C}$ に加熱することである。請求項15では、加熱した状態で圧縮成形を行い、離型された熱い圧粉体を大気中、不活性ガス中、減圧中の何れかにおいて、前記結合用樹脂の少なくとも1種が溶融する温度で加熱処理する態様、つまり加熱圧粉成形と圧粉体の加熱処理を連続して行うことを特定した。請求項16では、圧縮成形体を加熱処理した圧粉磁心を、温度150~320 $^{\circ}\text{C}$ で加熱して安定化熱処理を施す態様を特定した。

【0011】

【発明の実施の形態】次に、以上の発明を実施の形態及び実施例により明らかにする。この説明では、まず、発明対象の磁性粉末、結合用樹脂及びその含有量、磁性粉末と樹脂の混合、圧縮成形、加熱処理、安定化熱処理について順に詳述する。その後、試験の代表的なものを実施例に挙げ利点を明らかにする。

【0012】(1. 磁性粉末)対象の鉄粉は、アトマイズ法、還元法等の各種製法による鉄粉を適用することができる。鉄粉の粒度は、要求される磁束密度及び使用される周波数領域により選択される。粒子径は一般に粉末冶金で使用される200 μm 以下を使用することができるが、圧縮性を考慮すると150 μm 以下である。鉄粉

の粒子径が小さいほど過電流損失が小さくなり、高周波特性が向上するため、粒子径は100 μm 以下とすることがより好ましい。細かい粒子については特に限定しなくてもよいが、細かな粒子が多い粒度分布は、粉末圧縮性及び粉末流動性が悪くなり、高密度な圧粉磁心が得られないため、粒子径10 μm 以上の粉末とすることが好ましい。

【0013】磷酸化合物被膜を表面に施した鉄粉は、その被膜が絶縁層として作用し、鉄粉粒子間の過電流発生を抑制する効果があり、結合用樹脂の存在によって、過電流の発生を抑制する効果がさらに高くなり、より高周波特性が向上する。被膜用磷酸化合物は、磷酸鉄、磷酸マンガン、磷酸亜鉛、リン酸カルシウム等が好適である。また、磷酸化合物被膜を表面に施した鉄粉の市販品でも差し支えない。この例としては、ヘガネス社製の鉄粉(商品名:Permite、或いはSomaloy)等が挙げられる。

【0014】(2. 結合樹脂)結合用樹脂としては、耐熱性に優れるPPS、熱可塑性PIもよい特性を示し好適である。圧粉磁心の使用される温度が180 $^{\circ}\text{C}$ を越えるような環境であり、長時間にわたって使用されると、圧粉磁心の形状、寸法に経時変化を生じたり、見掛けの絶縁性能が低下する虞がある。その理由は、前者は圧縮成形時に生じる複雑な残留応力があるものと考えられること、後者は高温環境により、磁性粉末間の絶縁樹脂の厚さが減少する可能性が考えられる。このような虞には、前記PPS又は熱可塑性PIに、これらよりガラス転移点が高い樹脂を混合すると、特性が改善される。これは、磁性粒子(鉄粒子)間の樹脂が、熱特性が異なる複合状態であるため、使用中の変形や移動を生じ難くしているものと考えられる。ガラス転移点が高い樹脂の含有量は、主体となるPPS又は熱可塑性PIの量を超えない範囲とされる。PPSに熱可塑性PIを混合することも、この技術思想と同じことである。

【0015】(3. 結合樹脂の含有量)結合用樹脂の含有量は、全質量の0.15~1質量%の範囲が好適である。0.15質量%未満では、磁性粉末の粒子が結合及び絶縁する効果が少なく、圧粉磁心の強度が不十分となり、磁性粉末間の絶縁性が悪いものとなる。また、結合用樹脂の含有量が1質量%を越えると、圧粉磁心の強度及び絶縁性が高いものとなる反面、圧粉磁心に占める磁性粒子の占有率が低くなり、磁性粒子の密度が低いものとなるため、高い磁束密度及び透磁率が得られなくなる。

【0016】透磁率との関係において、50Hz程度の低周波数領域では樹脂の含有量が多いものほど低くなる。5kHz程度の高い周波数の領域では、樹脂を含まないものは低周波領域における透磁率よりはるかに低い値を示し、これに比べて、樹脂含有量が0.3質量%近傍で透磁率が最大値を示して低周波における透磁率とは

ほぼ同じ値となる。樹脂含有量がさらに増加すると、低周波の場合の透磁率と同様に、次第に低下していき、樹脂含有量が1質量%を越えると、樹脂を含まない場合の透磁率より低くなる。このような樹脂量と透磁率の関係からも、結合用樹脂の含有量は、0.15~1質量%が最適となる。樹脂含有量は、0.3質量%近傍が特に好ましい。また、密度は7.35g/cm³以上であることが望ましい。

【0017】(4. 磁性粉末と結合用樹脂の混合) 結合用樹脂は、磁性粉粒子間を絶縁し、過電流の発生を抑制する。磷酸化合物被膜を施した鉄粉は、粉末圧縮成形の際に剥離や脱落によって、磷酸化合物による絶縁が破られる虞があるが、結合用樹脂の存在によって保護され、より過電流の発生を抑制することができる。

【0018】結合用樹脂は粉末の形で混合することができる。その際は、磁性粉末の粒度分布と同等又は細かめとすると、混合状態が良好になり、耐熱性も向上する。磁性粉末間の絶縁性を向上させるためには、60μm以下の粒度とすることが好ましい。また、結合用樹脂に、n-メチル-2-ピロリドン等の極性が強い有機溶剤を添加して低粘度化させておき、流動層式又は攪拌混合式のコーティング装置を用いて、鉄粉に必要な量のコーティングを行ったのち、乾燥する方法も好ましい。

【0019】また、有機溶剤を含む結合用樹脂を、前記した場合より少ない樹脂量でコーティングしたのち乾燥して、樹脂被覆粉末を作り、この樹脂被覆粉末に結合用樹脂の粉末を混合する方法としてもよい。有機溶剤を含む結合用樹脂を用いて得られる樹脂被膜は、絶縁性能がより優れたものとなる。樹脂の膜厚は、20nm以上であると過電流の発生が少なくなる。膜厚20nmの樹脂被膜を得るには、おおよそ樹脂量0.15質量%程度となる樹脂溶液が混合される。一方、樹脂膜厚が200nmを越えると、粉末の圧縮性が悪くなり、その結果、磁気特性の不十分な圧粉磁心になる。樹脂を被覆した磁性粉末に、追加で結合用樹脂粉末を混合すると、被覆樹脂が保護され、より優れた磁性特性が得られる。

【0020】(5. 圧縮成形) 樹脂被覆磁性粉末は金型を用いて圧縮成形される。圧縮成形のとき、圧縮性向上や圧粉体抜き出し摩擦の低減のために、金型面に粉末冶金で通常用いられるステアリン酸亜鉛やエチレンビスステアロアミド等の成形潤滑剤粉末を静電塗布等により予め塗布しておくことが望ましい。また、より高い密度に成形するには、結合用樹脂が溶融しない温度に加熱した状態で行う態様、混合粉や樹脂被膜鉄粉を加熱しない状態で1次圧縮成形した後、結合用樹脂が溶融しない温度に加熱した状態で2次圧縮成形を行う態様、更に結合用樹脂が軟化する温度から溶融する温度まで加熱した状態で圧縮成形を行う態様で行うことである。なお、成形後処理としては、成形したのち、常温まで冷却して、以降に述べる加熱処理を行う方法としてもよいが、成形し

たのち、成形体が熱いままで加熱処理へ移行する方法とすれば熱エネルギーと冷却時間を省くことができるので、合理的である。

【0021】(6. 加熱処理) 加熱処理は、結合用樹脂を溶融させ、さらに結合用樹脂の結晶化による樹脂特性の安定化を図る工程である。加熱温度、加熱時間は使用する樹脂の種類により選定される。温度は、樹脂の融点から樹脂が熱劣化しない範囲であり、PPSでは250~400℃、熱可塑性PIでは300~450℃とされる。加熱時間は一般的に約0.5~1時間程度である。

【0022】加熱時の雰囲気は大気中で行うことができる。但し、大気中の酸素の存在は、樹脂の強度低下、機械的特性の低下を生じる虞が考えられる。これは、酸素の存在によって、樹脂の重合反応が進行し、ガス状の縮合物が発生しやすくなり、樹脂内に気泡として残存することも起こり得るからである。そのため、より好ましくは、大気中での加熱に先立ち、窒素ガス等の不活性ガス雰囲気中で加熱される。また、減圧された雰囲気中で加熱すると、雰囲気中の酸素量が減少すると共に、ガス状の縮合物をより樹脂から放出させることができる。これら雰囲気は、適宜組み合わせることができる。加熱処理の冷却過程では、温度320~150℃程度の領域において時間をかけて冷却すると、以下に述べる安定化熱処理を兼ねることができる。

【0023】(7. 安定化熱処理) 安定化熱処理を行うと、結合用樹脂の特性を安定化し、圧粉磁心を高い温度で使用したとき、経時変化を生じ難いものとすることができる。この場合、前記加熱処理を行い、一旦、冷却したのち、150~320℃程度で1~2時間程度加熱される。また、前記加熱処理の冷却過程で、320~150℃程度の温度領域で1~2時間保持する方法によることもできる。

【0024】

【実施例】次に、本発明の実施例と比較例により、発明構成及び利点を明らかにする。

(1) 準備した粉末は次の(1)~(8)の8種類である。

(1). アトマイズ鉄粉：これは「ヘガネス社製、品番：ABC100.30,」粒度が150μm以下のものである(以下、純鉄粉と記す)。

(2). 磷酸被膜処理アトマイズ鉄粉：これは「ヘガネス社製、品番：Somaloy500,」粒度が150μm以下のものである(以下、被膜形成鉄粉と記す)。

(3). 熱可塑性ポリアミド樹脂(以下、ポリアミドと記す)入り磷酸被膜処理アトマイズ鉄粉：これは「ヘガネス社製の市販粉末」、磷酸被膜処理アトマイズ鉄粉(Somaloy500)に熱可塑性ポリアミドを0.6質量%混合したものである。

(4). PPS粉末：これは「大日本インキ製」粒度が150μm以下(-150μm)のもの、60μm以下(-60μm)のものである(以下、PPSと記す)。

(5). 熱可塑性ポリイミド粉末:これは「三井化学製」、粒度が $150\mu\text{m}$ 以下 ($-150\mu\text{m}$) のものと、及び $60\mu\text{m}$ 以下 ($-60\mu\text{m}$) のものである(以下、熱可塑性PIと記す)。

(6). 熱硬化性ポリイミド粉末:これは「ローランヌ製」、粒度が $150\mu\text{m}$ 以下 ($-150\mu\text{m}$) のものである(以下、熱硬化性PIと記す)。

(7). 熱硬化性フェノール樹脂粉末:これは「大日本インキ製」、粒度が $150\mu\text{m}$ 以下 ($-150\mu\text{m}$) のものである(以下、フェノールと記す)。

(8). ステアリン酸亜鉛粉:これは一般に使用されている成形型用潤滑剤である。

【0025】(2)

【実施例1】樹脂の含有量と圧粉磁心の実効透磁率
前記被膜処理鉄粉にPPS ($-150\mu\text{m}$) を所定量(全質量%で、0%、0.15%、0.3%、4.5%、6.0%、7.5%、1.0%、1.2%となるよう)混合し、ステアリン酸亜鉛粉を塗布した金型を用い、混合粉を成形圧力 1470MPa でリング形状($\phi 10 \times \phi 23 \times 5\text{mm}$)に圧縮成形した。成形体は、空气中で温度 320°C で1時間加熱したのち、温度 240°C で1時間加熱して、冷却することにより圧粉磁心を得た。なお、PPSを含まないものは純圧粉体である。

【0026】実効透磁率は、B-Hアナライザーにより測定した。周波数は、 50Hz 及び 5000Hz で、印*

* 加磁束密度は 1T (テスラ) である。実効透磁率の測定結果は図1の通りである。即ち、 50Hz における実効透磁率は、樹脂含有量の増加に対してほぼ直線的に低下している。一方、 5000Hz における実効透磁率は、PPSを含まないものは低く、PPS含有量が0.3質量%近傍で最大値となり、それ以上のPPS含有量では、緩やかに低下しており、PPS含有量が1質量%のとき、PPSを含まない圧粉磁心の値とほぼ同じになっている。なお、樹脂がPPSの例を挙げたが、他の樹脂粉末の場合でも同様な傾向となる。

【0027】以上のことから、発明の樹脂含有量に関し、樹脂含有量の少ない領域では、実効透磁率の平均変化率が大きいので、樹脂含有量は0.15質量%以上とした。樹脂含有量の多い側では、樹脂を含まない圧粉磁心の実効透磁率より低くならない1.0質量%以下の樹脂含有量とした。

【0028】(3)

【実施例2】純鉄粉を用いた樹脂の種類と圧粉磁心の耐熱性

前記純鉄粉に各樹脂粉末を所定量添加し、V型混合機で混合した。樹脂粉末の混合割合は表2に示す通りである。PPS及び熱可塑性PIは粒度が $-150\mu\text{m}$ のものを用いた。

【表2】

試料 No.	樹脂粉末の種類と含有量 (質量%)				備考
	PPS	熱可塑性PI	熱硬化性PI	フェノール	
A1	0.15				発明
A2	0.6				"
A3	1.0				"
A4		0.6			"
A5	0.3		0.3		"
A6		0.3	0.3		"
A7				0.6	比較例

【0029】各混合粉は、成形圧力 1470MPa で円柱($\phi 23 \times 5\text{mm}$)及び円筒形状($\phi 10 \times \phi 23 \times 10\text{mm}$)に圧縮成形した。成形では、金型の内壁面に予めステアリン酸亜鉛を静電塗布しておき、混合粉を充填及び圧粉した。成形体の加熱処理温度は、樹脂がPPSを含むもの、及び熱可塑性PIを含むものは 320°C 、フェノールを含むものは 150°C とし、窒素ガス雰囲気中でそれぞれ1時間加熱した。安定化熱処理は、樹脂がPPS及び熱可塑性PIを含むものについて行い、大気中で、 240°C で1時間加熱した。

【0030】円柱形状($\phi 23 \times 5\text{mm}$)の各試料は、 $5 \times 23 \times 5\text{mm}$ の角柱形状に切削加工し、絶縁性能評

価に供した。絶縁性能評価は、温度 200°C の恒温層中で、100時間加熱したのち、四端子法(試料両端に直流電流を流し、その間に2端子を接して電気抵抗を測定)による見掛け固有抵抗値を測定し、加熱する前の値に対する低下率で評価した。また、円筒形状($\phi 10 \times \phi 23 \times 10\text{mm}$)の各試料については圧環強度値を測定した。圧環強度は室温及び温度 200°C において、圧縮速度毎分 0.5mm で圧縮して破壊するまでの最大荷重である。表3に見掛け固有抵抗、室温及び 200°C の圧環強度の測定結果を示した。

【表3】

試料 No.	見掛け固有抵抗 ($m\Omega \cdot cm$)			圧縮強度 (MPa)			備考
	初期値	加熱後	低下率 (%)	室温	200℃	低下率 (%)	
A1	2.0	1.1	45	185	150	19	発明
A2	5.0	3.5	30	110	80	27	〃
A3	7.0	5.7	19	75	50	33	〃
A4	5.0	3.5	30	110	90	18	〃
A5	5.0	4.3	14	110	80	27	〃
A6	5.0	4.5	10	110	85	23	〃
A7	7.0	1.2	83	90	30	67	比較例

【0031】（評価）見掛け固有抵抗は、樹脂の含有量の増加によって一次関数的に上昇する。樹脂含有量が0.15質量%と1質量%では見掛け固有抵抗が異なるが、圧粉磁心の用途によってそれぞれ実用できるものである。樹脂の種類による加熱前後の見掛け固有抵抗をみると、PPSを含むもの（試料A1～A3）は樹脂含有量が変わっても加熱前後の変化量がほぼ同じで、低下率（変化率）では樹脂含有量が多いほど少なくなっている。熱可塑性PIを含むもの（試料A4）でも同じになっている。PPSに熱硬化性PIを含むもの（試料A5）、及び熱可塑性PIに熱硬化性PIを含むもの（試料A6）は、見掛け固有抵抗の変化量は、PPSのものより少なくなっている。これらに比べて、フェノールを含むもの（試料A7）は、加熱前の見掛け固有抵抗はPPS等を含むものより高いが、加熱後の変化量がきわめて多く、加熱後の見掛け固有抵抗が著しく低いものとなっている。

【0032】圧縮強度は樹脂含有量が多いほど低くなる。室温と200℃との差は、PPSを含むもの（試料A1～A3）及び熱可塑性PIを含むもの（試料A4）共にほぼ同じであるが、フェノールを含むもの（試料A7）は、室温の強度も低い、200℃における強度が著しく低いものとなっている。

【0033】（4）

【実施例3】被膜形成鉄粉を用いた樹脂の種類と圧粉磁

心の耐熱性

前記被膜形成鉄粉に、表4に示す各樹脂を所定量添加した混合粉を作製した。なお、試料B16のポリアミドは上記した磷酸被膜処理アトマイズ鉄粉(Somaloy500)にポリアミドを0.6質量%混合した市販粉末である。試料B13は、PPSに有機溶剤としてn-メチル-2-ピロリドンを添加した液を被膜形成鉄粉に加えて混合し、乾燥してPPS含有量が0.15質量%で被覆された磁性粉末としたのち、更にPPSを混合してPPS含有量を0.6質量%とした混合粉である。それ以外の混合粉は、被膜形成鉄粉に樹脂の粉末を添加し、V型混合機で混合した。試料B14～16は比較例である。各混合粉は、前記実施例2と同様な条件で円柱（φ23×5mm）及び円筒形状（φ10×φ23×10mm）に圧縮成形した。

【0034】成形体の加熱処理温度は、樹脂がPPS及び熱可塑性PIを含むものは320℃、熱硬化性PIを含むものは200℃、フェノールを含むものは150℃、ポリアミドを含むものは275℃とし、窒素ガス雰囲気中でそれぞれ1時間加熱した。なお、試料B12については空気中で行った。安定化熱処理は、樹脂がPPS及び熱可塑性PIを含むものについて行い、温度240℃で1時間加熱した。

【0035】

【表4】

試料 No.	樹脂の種類と含有量 (質量%)						
	PPS -150	PPS -60	熱可塑性PI -150	熱可塑性PI -60	熱硬化性PI	フェノ ール	ポリ アミド
B1	0.15						
B2	0.3						
B3	0.6						
B4	1.0						
B5	0.1		0.05				
B6	0.3		0.3				
B7			0.6				
B8	0.3				0.3		
B9			0.3		0.3		
B10		0.6					
B11				0.6			
B12	0.6						
B13	0.6						
B14					0.6		
B15						0.6	
B16							0.6

【0036】前記実施例2と同様な方法で、温度200℃で100時間加熱したのちの見掛け固有抵抗値と、室温及び温度200℃における圧環強度を測定した。測定*

*結果は表5のと通りである。

【0037】

【表5】

試料 No.	見掛け固有抵抗(mΩ・cm)			圧環強度(MPa)			備考
	初期値	加熱後	低下率(%)	室温	200℃	低下率(%)	
B1	5.0	3.5	30	185	150	19	発明
B2	8.0	6.0	25	155	120	23	#
B3	12.0	10.0	17	110	80	27	#
B4	17.5	16.0	9	75	50	33	#
B5	5.5	3.5	30	185	150	19	#
B6	12.0	10.0	17	110	80	27	#
B7	12.0	10.0	17	110	95	14	#
B8	12.0	11.0	8	110	80	27	#
B9	12.0	11.5	4	110	80	27	#
B10	15.0	13.5	13	130	100	23	#
B11	15.0	13.0	10	110	90	18	#
B12	17.0	13.0	24	110	80	27	#
B13	13.0	12.0	8	110	85	23	#
B14	9.0	8.0	11	90	70	22	比較例
B15	10.0	4.0	60	90	30	67	#
B16	8.0	3.0	63	90	20	78	#

【0038】(評価)見掛け固有抵抗は、純鉄粉を用いた圧粉磁心より高い。磷酸化合物被膜があるだけ、鉄粉

粒子の絶縁がよくなっていることが分かる。樹脂の含有量の増加によって一次関数的に上昇することは、純鉄粉のものと同様である。樹脂の種類による加熱前後の見掛け固有抵抗をみると、PPSを含むものは樹脂含有量に係わらず、変化量がほぼ同じであり、低下率（変化率）では樹脂含有量が多いほど少なくなる。また、粒度が $-150\mu\text{m}$ のPPS、このPPSと熱可塑性PI又は熱硬化性PIとの混合物のもの、粒度が $-150\mu\text{m}$ の熱可塑性PI及びこの熱可塑性PIと熱硬化性PIとの混合のものは、ほぼ同じ特性を示すが、細部的にはPPS又は熱可塑性PIに熱硬化性PIが混ざっているものが、加熱による見掛け固有抵抗の低下が少ない。

【0039】PPSも熱可塑性PIも添加した粉末の粒度を $-60\mu\text{m}$ としたものは、加熱前後ともに粒度 $-150\mu\text{m}$ のものより高くなっている。試料B13のPPSを湿式混合して被覆し、PPSを混合したものは、粉末で混合したものより、僅かに見掛け固有抵抗が高い。試料B13の加熱処理を空気中で行ったものは、加熱による見掛け固有抵抗の低下が大きい、窒素ガス中加熱より高い値を示している。これらに比べて、フェノールを含むもの（試料B15）及びポリアミドのもの（試料B16）は、初期値が低く、加熱した低下量が大きくなっている。また、熱硬化性PIだけを含むもの（試料B16）では、加熱による低下量は少ないが、低い値を示している。

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*【0040】圧環強度は、純鉄粉の場合と殆ど同じ水準で、樹脂含有量との関係及び 200°C に加熱したときの低下量共に同じ傾向を示している。圧環強度においては、樹脂粉末の粒度の影響、樹脂の湿式被覆、加熱処理の雰囲気中の違いに差は認められない。PPS系及び熱可塑性PI系に比べて、フェノールを含むもの及びポリアミドのものは初期値が低く、加熱したときの低下量が大きいこと、及び熱硬化性PIが加熱による低下量は少ないが、低い値を示していることは、見掛け固有抵抗の場合の序列と同じになっている。

【0041】

【発明の効果】以上説明したように、本発明の圧粉磁心は、鉄粉、又は磷酸化合物被膜を表面に施した鉄粉に、PPS樹脂または熱可塑性PI樹脂、及びこれらとガラス転移温度が比較的高い樹脂との混合物の形で、0.15~1質量%含むものとしたことで、透磁率が高く、特に高周波領域で使用される場合に優れた特性を示し、また、温度が高い環境で使用される場合でも、固有抵抗及び耐熱強度が高いものであるから、用いられる装置の性能及び小型化に寄与でき、圧粉磁心の適用範囲を拡大することができる。

【図面の簡単な説明】

【図1】磷酸化合物被覆鉄粉をPPS樹脂で結合した圧粉磁心の樹脂含有量と実効透磁率の関係を示すグラフである。

【図1】

